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POTATO-STARCH GELS

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The strength of potato-starch gels decreased greatly as the pasting temperature was raised from 85° to 95° C. Gel strength increased linearly with concentration between 6 and 11%. A pH below 4 and above 9.5 materially weakened the gels. Heating the starch at 93° C. with 28% moisture before pasting retarded swelling, increased firmness and strength, and lessened clarity and tackiness. Sucrose at concentrations up to about 35% increased fluidity of hot pastes, increased gel strength, clarity, and stability at high and low pH, and decreased tackiness. At higher concentrations, granule swelling was largely inhibited, and a self-preserving, 65% sucrose starch gel was not preparable. Sucrose did not gel 8% starch dispersions in which the granules had been mechanically destroyed. Gel clarity was reduced much more by electrolytes than was gel strength. Potato starch prepared in the laboratory gave stronger and clearer gels than did commercial potato starch. The difference in gel strength was removed by washing the commercial starch with distilled water before pasting. The difference in clarity remained. The characteristic high initial clarity of potato-starch gels was lowered markedly on storage, particularly during the first 20 hours. Extent of granule swelling and persistence of granule sacs were correlated with gel strength.

LITTLE detailed information is available concerning potato-starch gels. The impression is fairly general that the gels are soft, elastic, and tacky, although some workers consider potato starch essentially nongelling (*6*). In contrast, the gelling power of corn and wheat starches is widely recognized, and a number of investigations on gels of these starches have been conducted (*4, 5, 7, 15*).

In the study of potato starch reported here, data on gel strength, firmness, and clarity, and on the effects of pH, temperature, and concentration were obtained. Properties of the gels were altered considerably by physical pretreatment of the starch and by addition of sucrose or electrolytes. The relation of gel properties to granule condition is shown in photomicrographs.

PROCEDURES

The great difference in pasting behavior between potato and cereal starches prevented adoption of techniques utilized in making gels of the latter (*4, 5, 15, 17*). Cereal starches form thin, pourable pastes that thicken slowly and set on cooling, but potato-starch pastes set quickly, and the hot paste viscosity of a gelling mixture is very high. The difficulty of gelatinizing uniformly, mixing, and heating such a paste is readily apparent. The necessity of preparing gels in an identical manner to ensure reproducible properties was shown in preliminary experiments. It was found that major changes in properties of starch gels may be associated with apparently minor and often unnoticed differences in their manner of preparation. The method ultimately adopted represents a modification of that employed by Ripperton (*10*).

The potato starches used in this work were a high grade commercial preparation and a laboratory sample in the preparation of which distilled water was the only agent used. The tapioca, wheat, corn, waxy corn, and waxy sorghum starches were commercial preparations.

Most starch pastes (300 grams each) were made by pipetting hot distilled water into a cold-water slurry of the starch. The slurry (2.5 parts of water, 1 part of starch) was placed in a 1-liter conical flask, which was swirled, then inclined, as the boiling water was delivered instantaneously from the pipet through a short wide-bore (1.2 cm.) rubber tube. By these means, mixing of the hot water and starch was completed (within about 1 second) before thickening of the paste occurred. If the flask were merely swirled and held upright as the hot water was added, the resulting paste tended to become more concentrated at the periphery of the flask. Inclining the flask and permitting the vigorous stream of hot water to tumble the starch over on itself led generally to the formation of a uniform paste. Any non-uniform paste was discarded.

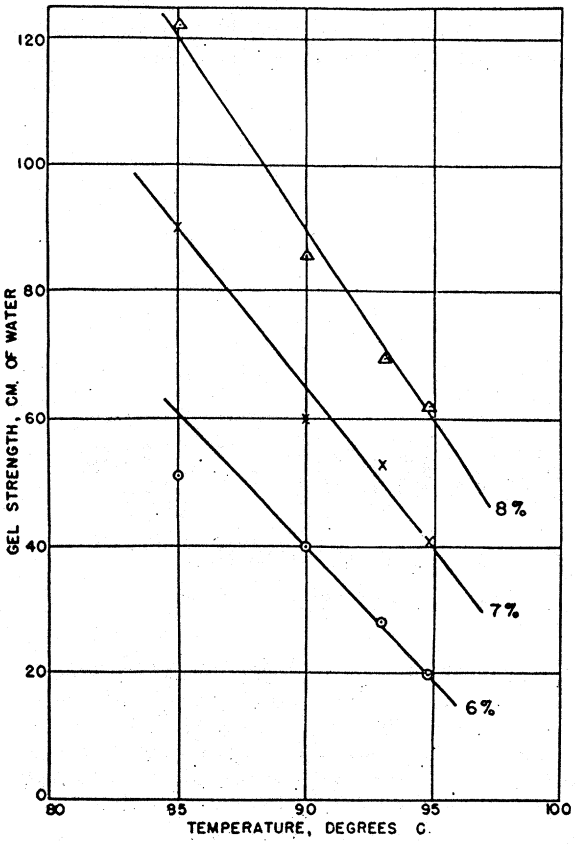


Figure 1. Effect of Pasting Temperature on Strength of 6, 7, and 8% Potato-Starch Gels

After the initial gelatinization (the temperature was about 75° C.), the flask was quickly transferred to a boiling water bath, and rotated at 17 r.p.m. in an inclined position for the required length of time or until a predetermined temperature was reached. Thus the paste was mixed gently by tumbling while pasting continued. Although mixing destroys a small proportion of the granules, it ensures temperature uniformity, which is much more important. In the concentrated pastes (10 to 12% starch), mixing and heat penetration were poor, as the pastes were too viscous to tumble. Usually the pastes were heated to 90° C. The time required to reach this temperature ranged from about 5.5 minutes with the 6% pastes to about 9.5 minutes with the 12% pastes. Modifications which decreased viscosity, such as heat-moisture treatment of the starch or addition of sucrose, also decreased the time needed to reach 90° C.

After the pastes reached 90° C., they were poured into 200-ml. jelly glasses. Any large air bubbles were removed with a glass rod. Separate portions of the paste were set aside for microscopic observation and measurement of light transmittance.

The jelly glasses were covered and stored at 25° C. for 22 hours. Gels were removed from the glasses and inverted before being tested. Gel strength was measured with a Delaware jelly tester (*1*). The piston was 15 mm. in diameter and was fitted with a stainless steel head 20 mm. in diameter, shaped as described by Mottern and Karr (*9*). Air flow was adjusted to raise the column of water 160 cm. in 60 seconds. Results are reported as centimeters of water pressure required for breaking the gel surface by the plunger. (In reporting gel strength, the convention of neglecting the pressure due to the weight of the plunger was followed. The weight of the plunger in its cylinder was equivalent to 17 cm. of water pressure.) A measure of gel firmness, comparable to the bulk modulus, was obtained by noting the pressure required to force the plunger into the gel a unit distance. pH measurements were made on a freshly exposed surface. Clarity of gels was determined by transmittance measurements with a Lumetron photoelectric colorimeter, and vertical cells were used that had an inside diameter of 19 mm., and red light of effective wave length of approximately 7500 Å. (tungsten lamp radiation, with Corning filters Blue Purple Ultra No. 5850 and Lantern Red No. 2412). Use of this long wave-length range made it possible to measure the transmittance of gels of relatively high opacity and at the same time avoided spurious transmittance effects due to pigments or impurities absorbing at shorter wave lengths. Clarity is reported as per cent transmittance compared with water.

To make the photomicrographs, usually 18 ml. of hot water were added slowly, with intermittent and gentle stirring, to 2 grams of the hot starch paste. Part of this diluted paste was mounted in a Howard mold-counting chamber 0.1 mm. deep. Thus a film of uniform thickness was obtained, and quantitative comparisons were made on granule sacs among the various samples. No staining was employed, as most dyes were ineffective, and iodine–potassium iodide solution greatly altered the character of the paste. Although the granule sacs were scarcely visible under ordinary direct illumination, they could be seen and photographed readily if an opaque disk with a slot cut in one side was placed immediately below the condenser. This device prevented the entrance of direct light but permitted lateral, shadow-casting illumination.

RESULTS

EFFECT OF TEMPERATURE. Potato-starch gel strength was dependent in large measure upon the temperature to which the paste was heated, as is shown in Figure 1. Raising the temperature from 85° to 95° C. decreased the gel strength by half, irrespective of starch concentration. Additional pasting at 95° C. lowered the gel strength still further. These results differ from those of Woodruff and Nicoli (*16*), who reported that heating the pastes to 90°, 95°, and 99.5° C. gave indistinguishable gels. However, they made no quantitative measurements of gel properties.

Although the gels when first made were almost water-clear to the unaided eye, under the microscope considerable structural detail became apparent. Clearly visible were innumerable granule sacs, folded, intertwined, and forming a continuous network throughout the gel. In an undiluted gel, the sacs were too thoroughly compacted to be separately distinguishable. Upon dilution, however, individual sacs, as well as others greatly entangled, were discernible. In the gel pasted to 85° C., the sacs were prominent and persistent (Figure 2, *A*); when the starch was pasted to 94° C., the sac membranes became thinner and less prominent, as indicated in Figure 2, *C*. Thus there was a relationship between the persistence of the granule sacs and gel strength. Brimhall and Hixon (*2*) have correlated the decrease in rigidity of cornstarch pastes with weakening of the granule membranes, and Cox and MacMasters (*3*) have shown by micromanipulation of individual potato-starch granules that the extensibility of the membranes is greater in samples pasted to 90° C. than in samples pasted to 80° C.

DEPENDENCE OF GEL PROPERTIES ON CONCENTRATION. Within the concentration range of 6 to 11% of starch, and with pasting to 90° C., gel strength varied directly with starch concentration (Figure 3). Uniform gels of 10% or greater starch concentration were prepared with difficulty, and the data on these gels showed greater fluctuations than those on the less concentrated gels. Vertical lines through the experimental points in Figure 3 indicate by their length the standard error of the arithmetic mean. Below 6% concentration, the pastes did not gel, but further experiments showed that gels could be prepared from pastes of less than 6% concentration, provided that the pasting temperature was

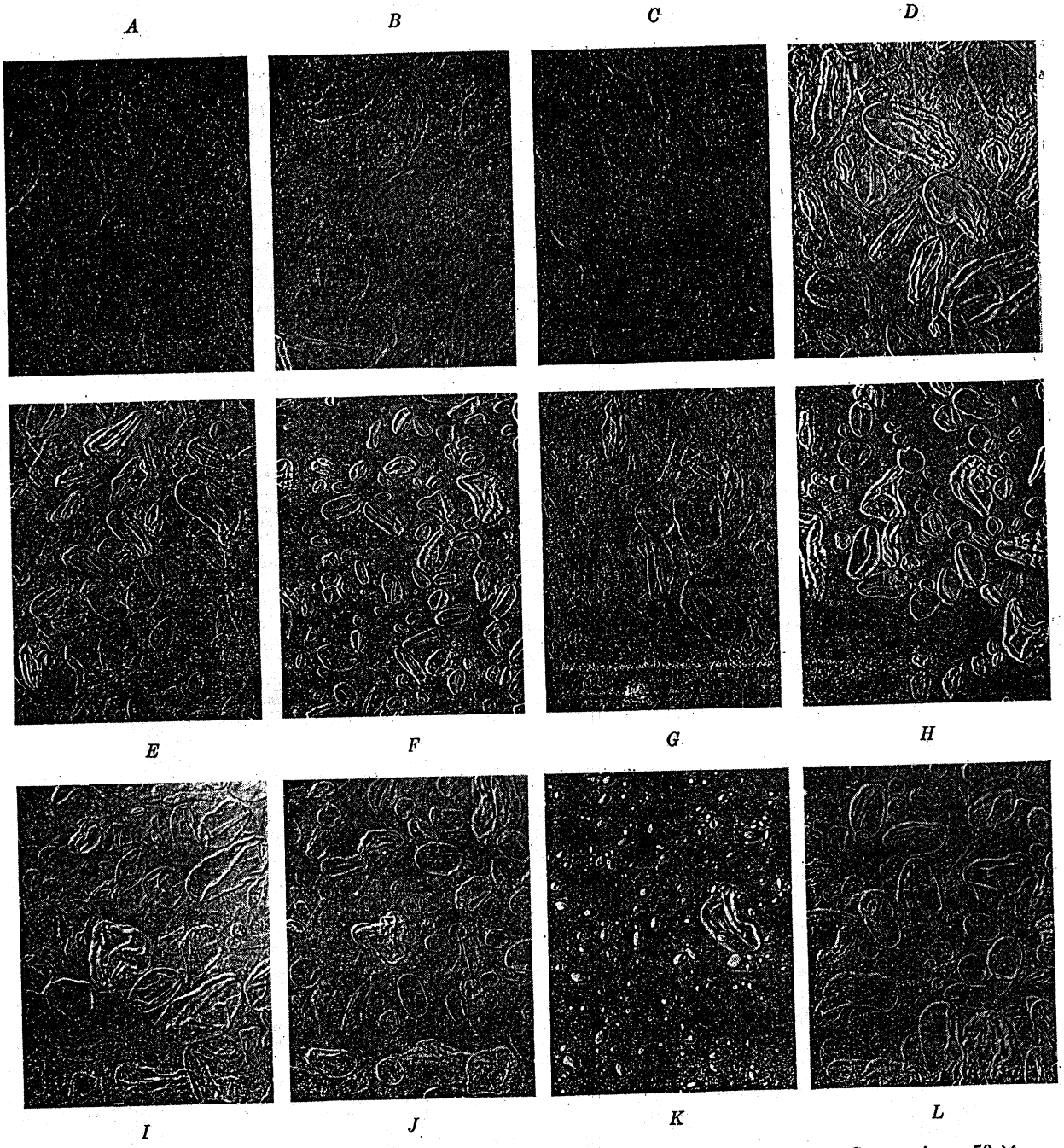


Figure 2. Photomicrographs of Potato Starch, Uniformly Diluted for Quantitative Comparison, 50 ×

Starch–water ratio on pasting, 8 to 92. Mixtures pasted to 90° C., except *A* and *C*

A. Starch pasted to 85° C.
B. Starch pasted to 90° C.
C. Starch pasted to 94° C.
D. Laboratory preparation
E. Heat-moisture treated for 2 hours
F. Heat-moisture treated for 8 hours
G. 6.8% sodium chloride, 7.5% starch
H. 33% dextrose, 5.3% starch
I. 23% sucrose, 6.2% starch
J. 33% sucrose, 5.3% starch
K. 65% sucrose, 2.8% starch
L. 33% sucrose, 5.3% starch, pH 3.5

tween 80° and 85° C. Other characteristics of the gels were changed but slightly by increasing the starch concentration; even the most concentrated gel was ropy and tacky. Firmness was only slightly greater in the concentrated gels. Translucency decreased with increasing starch concentration (Figure 4, curves 3, 4, and 6).

Effect of pH. With 8% gels pasted to 90° C., the effect on gel strength of varying the pH (by means of sodium hydroxide and hydrochloric acid) between 4 and 9 was slight (Figure 5, *A*). The minor increase in strength at pH 5 over the strength of the control (pH 6.1) possibly reflects an electroviscous effect. At pH values below 4 and above 9.5 there was considerable decrease in gel strength.

Translucency, firmness, and tackiness of the gels were also influenced by pH. Maximum translucency was exhibited between pH 6.5 and 7.5 (Figure 6, curves 4, 7, and 8). Below pH 6, translucency decreased rapidly. Above pH 8, the decrease was more gradual. Gel firmness, paralleling gel strength, declined steadily below pH 4 and above pH 9.5. Although most gels were tacky and exhibited rough surfaces upon removal from the jelly glasses,

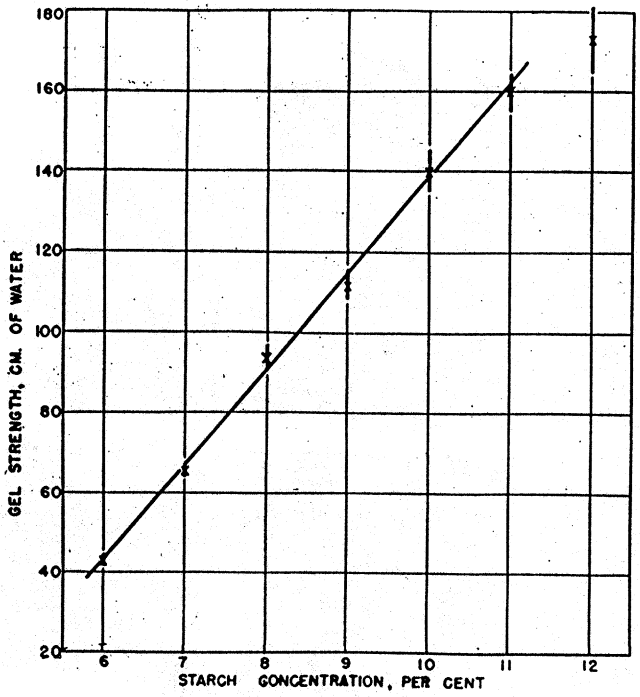


Figure 3. Dependence of Gel Strength on Concentration

Starch pasted to 90° C. Length of vertical lines indicates standard error of arithmetic mean of measurements.

there was one pH range in which this characteristic was altered. Below pH 5, the gels did not stick to their molds and presented smooth surfaces (Figures 7, *A* and *B*, and 12, *M*).

HEAT-MOISTURE TREATMENT. Sair and Fetzer (*11*) and Sair and Hilbert (*12*) have shown that heating potato starch at high humidity greatly alters its physical properties. Following their procedure, the authors heated potato starch at 93° C. with 28% moisture, with the results shown in Figures 8 and 9. Pretreating the starch for periods ranging from 1 to 8 hours greatly enhanced gel strength. Maximum strength was produced by a pretreatment of about 4 hours. The results shown in Figure 8 apply only to gels made under the conditions specified (8% starch pasted to 90° C.). Further pasting to 95 °C. decreased the gel strength of the 1-hour pretreated starch, but increased the strength of the 8-hour pretreated sample.

Heat-moisture pretreatment induced other significant changes in the physical properties of the gels. Firmness was greatly increased on prolonged treatment, approximating that of cornstarch gels (Figure 10). On the other hand, transmittance dropped markedly, and the gels were rather opaque (Figure 6, curves 4 and 9). Tackiness was lessened, and gel surfaces appeared smooth and glazed. In general, the effect of heat-moisture pretreatment was to confer upon potato starch the physical properties of a cereal starch.

Photomicrographs (Figure 2, *B*, *E*, and *F*) revealed that the changes in gel properties were correlated with changes in starch granule organization induced by the heat-moisture pretreatment. After pasting, pretreated granules were tougher and less swollen than those of the control, and fewer were broken. A moderate pretreatment, producing granules which swelled to an intermediate extent and very few of which burst, gave gels of maximum strength.

EFFECT OF HYDROGEN-BONDING AGENTS. Sucrose, dextrose, and glycerol incorporated into potato-starch pastes affected gel properties in a roughly parallel way. In all experiments, the proportion of starch to water was constant, 8 parts of starch to 92 parts of water. When sucrose was added to the 8% pastes, the gel strength changed, as shown in Figure 11. It rose to a maximum almost twice the initial strength when 50 grams of sucrose were added to 100 grams of 8% paste, then declined to an unmeasurably small value for equal weights of sucrose and paste. No increase in gel strength on addition of sucrose had been noted in the experiments of Woodruff and Nicoli (*16*) and Woodruff and McMasters (*15*). Gel firmness increased slightly when the pro-

TABLE I. STRENGTH AND TRANSMITTANCE OF 8% GELS

Item No.	Material[a] and Treatment	Gel Strength[b], Cm. H_2O	Transmittance[c], % 2 hr.	23 hr.	48 hr.
1	Potato starch, pH 6.2	90	80	34	24
2	Potato starch, washed 4 times with distilled water, pH 5.9	110	79	37	27
3	Potato starch, laboratory preparation, pH 6.0	102	..	53	47
4	Potato starch pasted with 0.01 *N* $CaCl_2$, pH 6.0	56	76	10	9
5	Potato starch pasted with 0.01 *N* NaCl, pH 6.2	55	76	10	9
6	Potato starch pasted with 0.01 *N* NaCl, pH 4.7	60	70	9	8
7	Potato starch, pH 4.7	103	67	9	7
8	Potato starch, with 20% glycerol, pH 6.4	155	76	22	18
9	Cornstarch, pH 5.0	173	2	1	1
10	Wheat starch, pH 5.9	123	4	2	1
11	Waxy cornstarch, pH 5.6	0	18	16	15
12	Waxy sorghum starch, pH 5.0	0	4	3	3
13	Tapioca starch, East Indian, pH 6.0	0	70	70	63
14	Tapioca flour, Brazilian, pH 5.9	0	31	23	18
15	Pectin, apple, 0.3% in 65% sucrose	...	79	77	79
16	Pectin, citrus, 0.4% in 65% sucrose	...	81	80	81
17	Gelatin, U.S.P., 8%	24	94	94	94

[a] All materials were commercial products except No. 3, a laboratory preparation.
[b] Measured after 22 hours' storage at 25° C.
[c] Measured after storage at 25° C. Light filters used were Corning No. 5850 (Blue Purple Ultra) and Corning No. 2412 (H. R. Lantern Red).

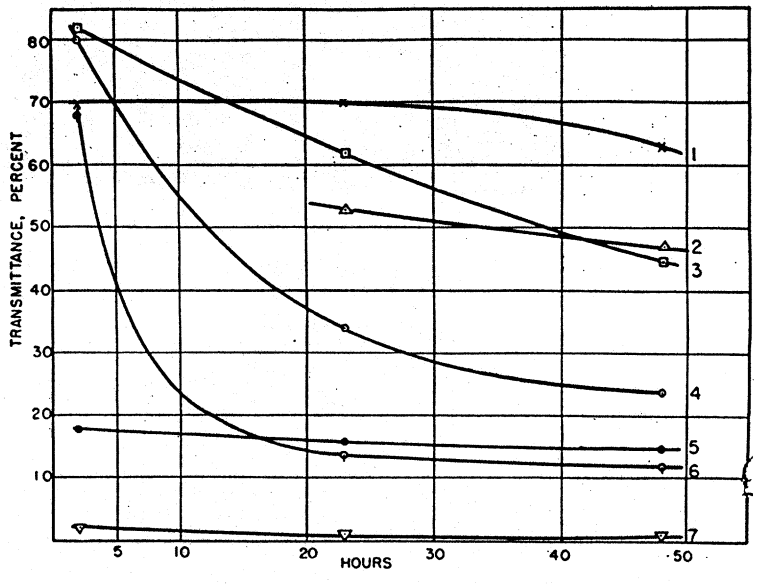


Figure 4. Dependence of Transmittance on Time

1. Tapioca starch, 8%
2. Potato starch, laboratory preparation, 8%
3. Potato starch, commercial, 6%
4. Potato starch, commercial, 8%
5. Waxy cornstarch, 8%
6. Potato starch, commercial, 10%
7. Cornstarch, 8%

portion of sucrose was high (Figure 10, curves 3 and 5). A hot paste containing 23% sucrose was thin and easily poured, but on cooling it gelled quickly, and was almost free of the tackiness characteristic of potato-starch gels. Figure 12 shows photographs of gels containing sucrose.

Comparison of *I*, *J*, and *K* with *E* and *F* of Figure 2 shows that a high concentration of sucrose had much the same effect on the starch granules during pasting as did heat-moisture pretreatment. In a paste containing 33% sucrose (Figure 2, *J*), the granules were only moderately swollen, and the sacs were prominent and strongly persistent. The gelatinization temperature was raised, and thickening of the paste occurred only after some time in the boiling water bath. In a 65% sucrose mixture, there was almost no granule swelling (Figure 2, *K*), even when the suspension was maintained at 95° C. for 20 minutes. It is evident that the sucrose molecules compete successfully with the starch molecules for the available water, leaving insufficient water for the swelling and gelatinization of the granules. This was conjectured but not proved by Woodruff and Nicoli (*16*). Figure 2, *H*, shows the extent of swelling of starch granules in 33% dextrose.

It was suspected originally that sucrose might be made to promote gelling of starch by a mechanism analogous to that operating in conventional pectin gels (*13*). In the experiments described above, possible hydrogen-bonding effects of sucrose upon starch molecules were obscured by the persistence of the granule sacs. Accordingly, starch-sucrose gels were prepared in which all microscopically visible granule structure was destroyed. An 8% starch suspension was gelatinized and then subjected at 75° C. to high mechanical shear. Amounts of sucrose sufficient to make the sucrose concentration 29, 38, 50, and 65% were added to portions of this paste, which were then mixed and heated to 90° C. The properties of the resulting samples indicated not only that sucrose was ineffective as a hydrogen-bonding agent for starch but that the persistence of granule sacs was necessary for forming starch gels, at least at starch concentrations as high as those specified. None of the samples set to a gel. All, together with a control to

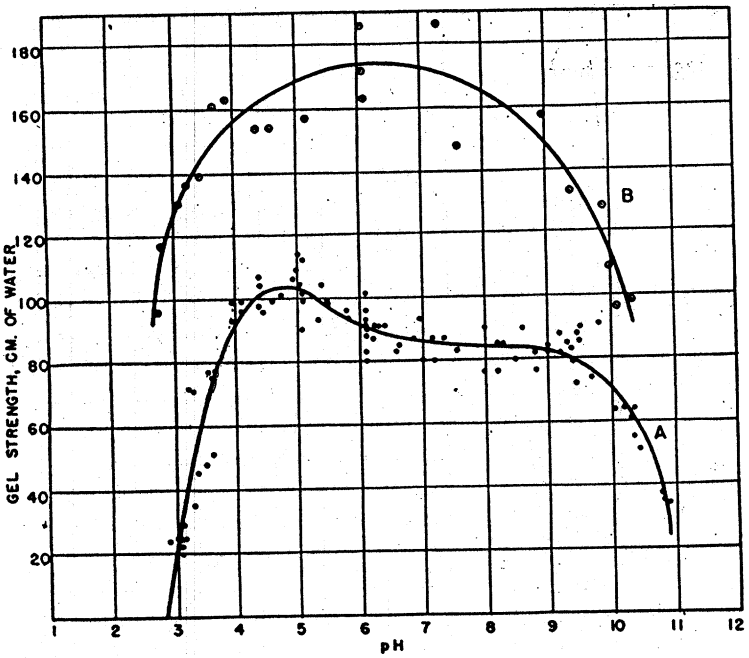


Figure 5. Variation of Gel Strength with pH

A. 8% potato starch
B. Potato-starch gels containing 33% sucrose (50 grams of sucrose added per 100 grams of 8% starch)

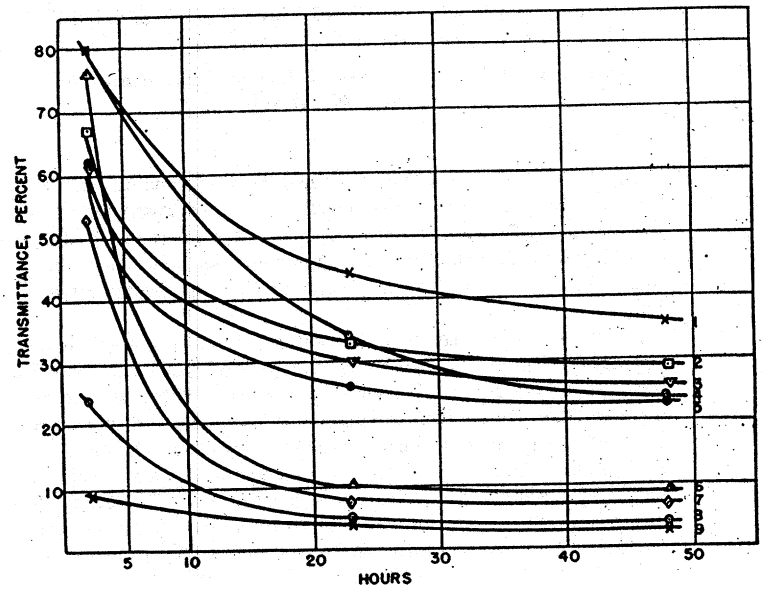


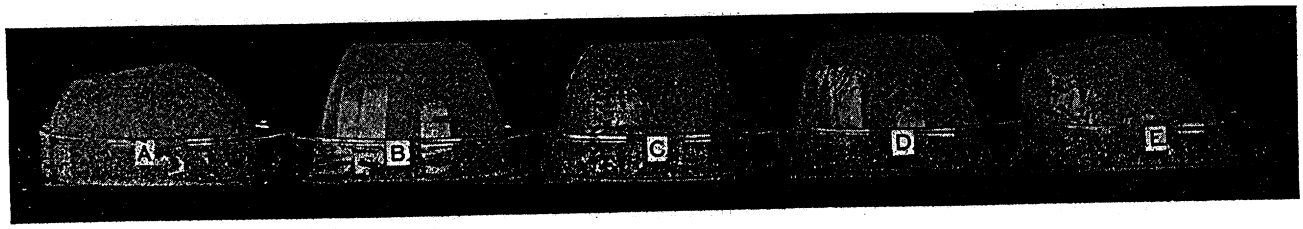
Figure 6. Dependence of Transmittance of Potato-Starch Gels on Time

All gels contained 8 parts of starch to 92 parts of water

1. 23% sucrose, 6.2% starch
2. 33% sucrose, 5.3% starch, pH 6.2
3. 33% sucrose, 5.3% starch, pH 3.1
4. 8% starch control, pH 6.1
5. 33% sucrose, 5.3% starch, pH 9.9
6. Gel containing 0.01 *N* calcium chloride
7. Gel pH, 9.8
8. Gel pH, 3.1
9. Starch heat-moisture treated for 4 hours

Figure 7. 8% Potato-Starch Gels

A. pH 3.1
B. pH 4.8
C. pH 6.3
D. pH 8.8
E. pH 10.5



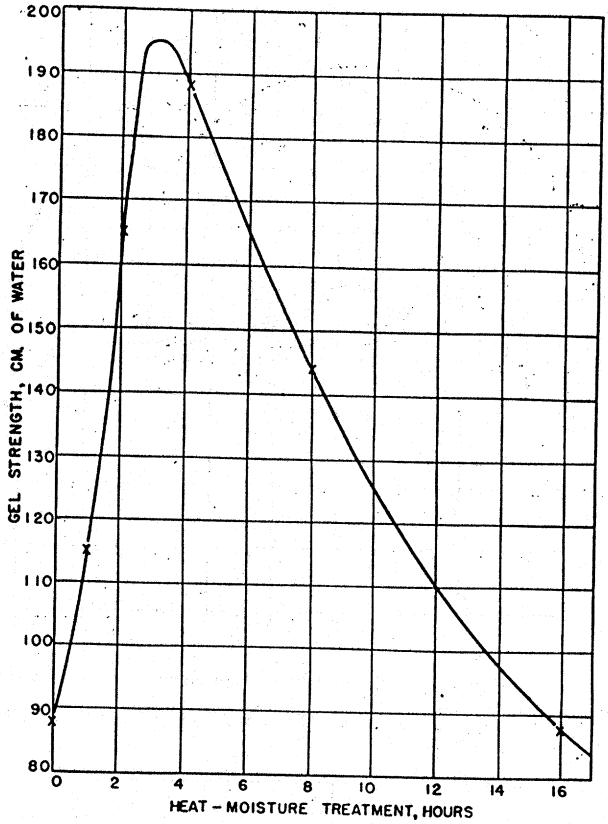


Figure 8. Effect of Heat-Moisture Treatment on Strength of 8% Gels

which no sucrose was added, remained fluid, viscous pastes. In contrast, firm gels can be made easily from a pectin-sucrose solution of only 1% pectin concentration. Other experiments indicated that neither dextrose nor glycerol is an effective gelling agent for potato starch in molecular or quasi-molecular dispersion.

The effect of sucrose on the transmittance of starch gels is illustrated in Figure 6. Two hours after pasting, both a starch gel containing 23% sucrose (curve 1) and the control (curve 4) gave the same high transmittance value of 80%. Transmittance of a 33% sucrose gel of the same age was 67% (curve 2). On standing for 23 hours, the gel containing 23% sucrose was much the clearest. The transmittance values had decreased to 44% for the 23% sucrose gel, 34 for the control, and 33 for the 33% sucrose gel. After 48 hours the values were 36, 24, and 29%, respectively. Table I (items 15, 16, and 17) shows by contrast that gelatin and pectin gels maintain their initial high transmittance unchanged over the same period. The decline in transmittance is an indication that molecules or molecule segments have aggregated into regions of high refractive index and correspondingly high scattering power. The clarifying effect of sucrose at a concentration of about 20% is attributed principally to the moderately increased viscosity and refractive index of the sucrose solution as compared to water. Increased viscosity retards molecular motion, and increased refractive index of the medium diminishes the turbidity arising from such aggregates as are formed. At sucrose concentrations of 33% (curve 2) and higher, inhibition of granule swelling results in a generally lower transmittance than is shown by an aqueous starch dispersion alone.

Apart from its effect upon gel strength, gelatinization tempera-

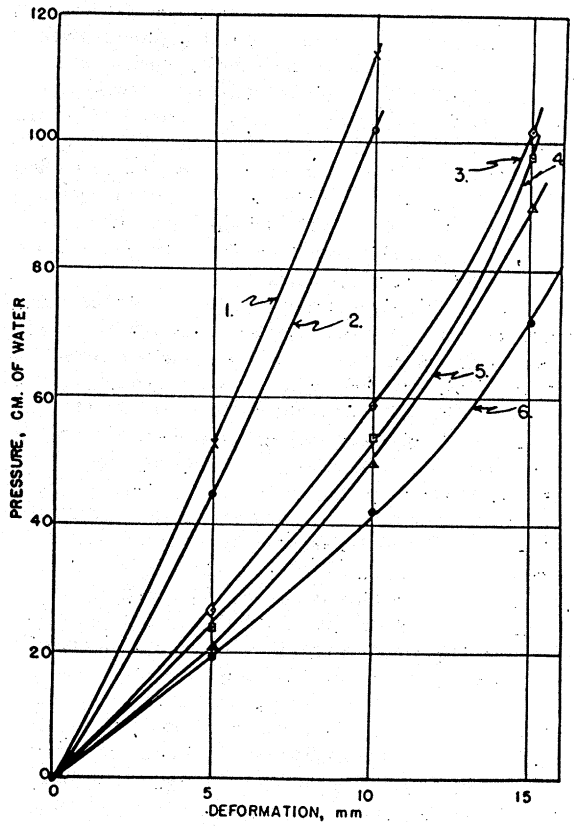


Figure 10. Firmness of 8% Starch Gels

1. Potato starch, heat-moisture treated for 8 hours
2. Cornstarch
3. Potato starch with 60 grams of sucrose added per 100 grams of 8% starch
4. Potato starch, heat-moisture treated for 2 hours
5. Potato starch, commercial, control
6. Potato starch, laboratory preparation

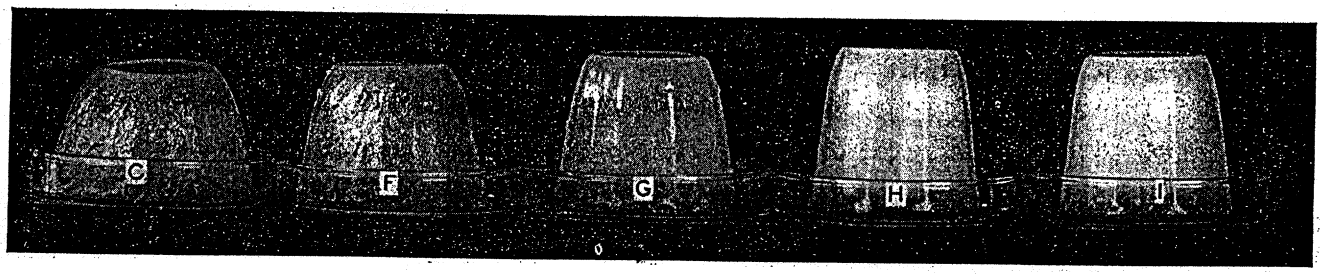


Figure 9. 8% Starch Gels

C. Potato starch control
F. Potato starch, heat-moisture treated for 1 hour
G. Potato starch, heat-moisture treated for 2 hours
H. Potato starch, heat-moisture treated for 8 hours
I. Cornstarch

ture, and clarity, sucrose has a protective action against acid and alkali impairment of potato-starch gels. Figure 5, *B*, shows the extent to which gel strength is increased by sucrose in the pH interval 2.8 to 10, and Figures 12, *M*, and 7, *A*, present photographs of gels with and without sucrose at pH 3.1. A microscopic study correlated the protective effect of sucrose with its capacity to prevent breakdown of the granule sacs during pasting. Sacs remained prominent and only moderately swollen even at pH 3.5 (Figure 2, *L*). The decided opacity of gels at pH 3.1 and 9.8 (Figure 6, curves 8 and 7) was diminished greatly in a 33% sucrose medium (curves 3 and 5).

EFFECT OF ELECTROLYTES. In comparison with the hot paste viscosity (*8*, *10*, *14*), the strength of potato-starch gels is little influenced by the mode of preparation of the starch or the electrolyte content of the water used in pasting. Items 1, 2, and 3 of Table I show a gel strength of 90 cm. for the commercial starch pasted with distilled water, 110 cm. for the same starch washed 4 times with distilled water, and 102 cm. for a laboratory starch processed only with distilled water. Pasting the commercial starch with either 0.01 *N* calcium chloride (200 p.p.m. of calcium) or 0.01 *N* sodium chloride (230 p.p.m. of sodium) reduced the gel strength to about 55 cm. (items 4 and 5). Transmittance was more sensitive to conditions of preparation and pasting. Transmittance values for the laboratory starch, washed commercial starch, commercial starch, and commercial starch pasted with 0.01 *N* calcium chloride, measured after 23 hours were, respectively, 53, 37, 34, and 10% (Table I and Figures 4 and 6). The laboratory starch paste was so thick that included air bubbles prevented the usual transmittance measurement after 2 hours. Gels made from laboratory starch were distinguished also by their ease of deformation (Figure 10, curve 6) and their relatively slight tackiness. Figure 2, *G*, is a photomicrograph of starch pasted in 6.8% sodium chloride.

OTHER STARCHES. A limited number of data on the gelling characteristics of other starches have been assembled in Figure 4 and Table I. Pasting conditions were the same as those selected for the measurements on potato starch—8% concentration, with heating to 90° C. East Indian tapioca starch gave a soft paste, too weak to be measured with the Delaware jelly tester. Its clarity was much the highest of any starch (70% at 2 hours) and decreased relatively little with time. The clarity of Brazilian tapioca flour, however, was reduced (Table I). Waxy corn and waxy sorghum starches also formed unmeasurably weak gels, but these had low transmittance (18 and 4% at 2 hours) despite their freedom from amylose, the rapidly precipitating component of most starches. Corn and wheat starches gave very strong, firm, opaque gels.

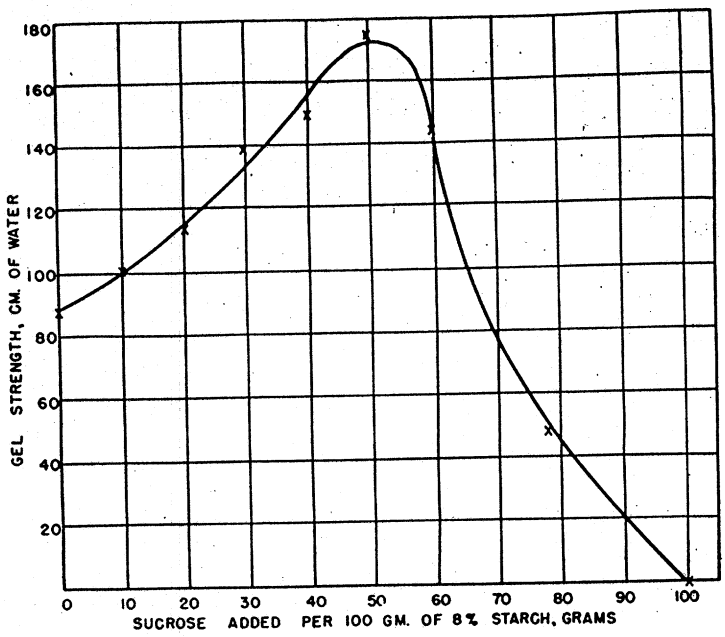


Figure 11. Effect of Sucrose on Gel Strength

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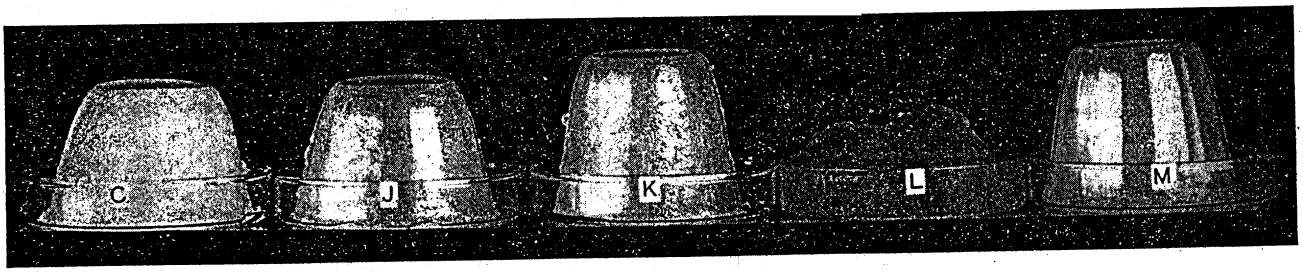

Figure 12. Potato-Starch Gels

All gels contained 8 parts of starch to 92 parts of water

C. Control, 8%
J. 23% sucrose, 6.2% starch
K. 33% sucrose, 5.3% starch, pH 6.2
L. 50% sucrose, 4.0% starch
M. 33% sucrose, 5.3% starch, pH 3.1